

# **Training Graduate Students in Utilization of Analytical Instruments in a Failure Analysis Course**

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## **Abstract**

Department of Engineering Technology at University of North Texas offers a graduate course on failure analysis (MSET 5150) during spring semesters. Partial requirement for the course is for students to submit a term paper based on their collected data related to a term project. Case studies are given to groups of students to work on actual failed components received from area industries. Results of their findings are presented at the end of the semester in both oral presentation form and written term paper form followed the format of a well-established technical papers. Results of such exercises allows graduate students devote skills in using scientific instruments and practice manuscript preparations for publication. This paper presents examples of a case studies done by groups of students who worked on failure analysis of components failed in an oil and gas industry. Students developed skills in utilization of scanning electron microscope (SEM), Energy Dispersive Spectroscopy (EDS), and Fourier Transform Infrared Spectrophotometry. This exercise has proven highly effective in introducing young engineers to real world problems in oil and gas industry and help them develop skills needed in performing failure analysis and steps involved.

## **1. Introduction**

In an undergraduate level most Engineering and Engineering Technology program cover only one course on Materials Engineering along with its corresponding laboratory. The coverage in those courses is limited to basic topics such as analysis of crystal structures, mechanical properties of ferrous and non-ferrous alloys, phase diagrams of binary systems, heat treatment of engineering alloys along with a chapter or two on each polymer and ceramic materials. Due to time limitations often times such courses do not include important topics such as failure analysis and corrosion. Department of Engineering

Technology at University of North Texas offers a sequence of graduate course one on Failure Analysis in the Fall semesters and another one on Corrosion every Spring semesters. Students who previously experienced the courses indicated their utmost satisfactions on the content and delivery of the subject matters. Some technical products of such courses are published in technical journals [1-6]. Failure analysis involves a number of steps such as mechanical testing, non-destructive testing etc. that are covered in depth in most undergraduate programs. However, there are some other advanced topics of such as utilization of analytical systems such as scanning electron microscopy, elemental analysis techniques such as energy dispersive spectroscopy, atomic absorption, and phase analysis such as x-ray diffraction (XRD) and Fourier transform infrared spectrophotometry (FTIR) which are not discussed in undergraduate programs. Advanced topics covering fundamentals of each technique, origin of data, interpretation and analysis of the spectrum, limitations of each technique such as detection limit, spectra interferences, and use of data are covered in the course. First author has contributed a chapter in a handbook of failure analysis in oil and gas industry [7].

In an effort to make the courses content most relevant to real life case studies, collaboration with a local industry was established in the last offering of the course in obtaining a number of failed engineering components in highly corrosive environments were obtained and assigned to each group of graduate students. Results were shared with industry representative for his company benefit in avoidance or minimization of such failures.

This paper reports on a selected sample data produced and analyzed by student groups in the failure analysis course in the Spring 2017 semester. Three cases are reported. Each case described in details and show students familiarity with analytical techniques utilized.

## 2. Case Study I: Corrosion of a Weir Metal Plate

There is an extensive variety of equipment and machinery used in the oil extraction industry. When well fluids are extracted from the ground they are contaminated mixed with water and other hydrocarbon products which affect the quality of the oil. Filtration is needed to separate the oil from other contaminants and water [8]. There are multiple filtration systems in the oil industry that utilize the primary methods of gravity settling, enhanced-gravity settling, coalescing, centrifugal force and electrical charge. For liquid-gas separators, one of the most common systems is the heated horizontal separator. Heat is added to assist the separation of oil and water as well as for protection of equipment. The basic components of horizontal heated separators include an inlet diverter, mist eliminators/coaleser, relief valves, pressure regulator, level control valves, water and inlet outlets, and a weir plate [9]. Fig. 1 shows the basic components and schematic of the separator. This type of separator uses a coaleser/settler method for separation. As the contaminated oil fluid enters the separator tank it fills up the drum and the water separates to the bottom of the tank allowing the oil to layer on top of the water. A weir plate allows the lighter oil to spill into another area of the tank where the oil outlet is located. Different literatures suggest these systems can handle pressures up to 2000 psi depending on the vessel size, temperatures  $-20^{\circ}\text{F}$ - $200^{\circ}\text{F}$  and materials used for the manufacturing of the internal components are typically carbon steel [9]. The weir is a metal plate usually quarter inch thick and welded to the bottom of the tank. A failed weir plate, shown in Fig. 2, of was analyzed by a variety of methods to identify cause of failure. It can be easily observed that corrosion was the main reason for failure making this failure mode the main focus of analysis was the main form of analysis. There are other factors that could have affected or increase the rate of corrosion thus flow actuated corrosion will be discussed during the conclusion section as well. Fig. 2 shows two feet section of a severely corroded weir plate showing uniform corrosion with extensive amount of corrosion products containing iron oxide ( $\text{Fe}_2\text{O}_3$ ) and oxy-hydroxides of iron ( $\alpha\text{-FeOOH}$ ,  $\gamma\text{-FeOOH}$ ) as identified by FTIR analysis shown in Fig. 3. Optical microstructure of the weir plate shows pearlite phase (Fig. 4.a) and SEM image showing uniform corrosion product morphology is shown in Fig. 4.b.

## 3. Case Study II: Hammer Union Corrosion

A Hammer Union is a pipe connection designed to be loosened or tightened by hammer blows on protrusions known as hammer wings used in a wide variety of harsh and viscous oil and gas exploration and drilling applications both onshore and offshore. Used on high pressure lines where quick disconnections are necessary.

Hammer union system include a sub and a nut as shown in Fig. 5 (a). Sub is made of low carbon steel with microstructure containing pearlite (Fig. 5.b) and the nut is nodular cast iron (Fig. 5.c). Corrosion of Hammer union (external, internal, or atmospheric) will depend on the environment and the changes that occur during the life of that Hammer union. The material used in the design of this component include

Uniform corrosion of hammer union exposed to corrosive environment in oil and gas industry was characterized for the form of corrosion.

A group of two students worked on the Hammer union system corrosion and utilized SEM and optical microscopes to observe morphological features of the corrosion mechanism and characterized corrosion products using FTIR and EDS. Their findings showed uniform corrosion as the main form of corrosion along with possibility of erosion corrosion due to high velocity corrosive agents flow. Through this project students reviewed advantages and disadvantages of engineering alloys such as cast iron and low carbon steel.

The extra thickness will provide extra material and make up for the corroded metal thus expanding the lifetime of the weir plate. Special coatings can be used reduce localized corrosion. New materials and coatings are arising. Fiber glass coatings have been proven effective for vessels operating at low pressure. It is important to maintain and uptake these systems.

## 4. Case Study III: Corrosion of API 5L, Grade B Carbon Steel Pipe

The operating conditions for the pipe segment are: pressure is less than 15 psi, fluid velocity is 300 BBL/Day which is low, fluid temperature is between  $150^{\circ}\text{F}$  ( $65.5^{\circ}\text{C}$ ). Also, the sample may be subjected to bacterial attack. As indicated in external part of the pipe in Figure 6 a, the pipe has been covered with an insulation or wrapped with a cover where large perforation was detected. Interior part of the part shows uniform corrosion. Usually when there is a highly localized failure such as the one shown in Fig. 6.a one possibility is stray form of corrosion due to unintended anode to cathode connection in underground piping system. In this case, since the pipe was used above ground the bacteria attack may have been the culprit for the failure. Generally bacteria and other microorganisms accumulate in a highly localized regions increasing acidity of the location by decreasing the pH. Low pH electrolytes severely attacks the alloy resulting in speedy dissolution of the metal leading to formation of large perforations such as the one shown in Fig. 6.a. Fig 6.b shows accumulations of corrosion products and other precipitates from the moving corrosive fluids.

## 5. Discussion

Trend in teaching and training graduate students in an Engineering Technology program requires courses that are practical in nature and provides students with a skill set that makes them useful for today's industry. Highly competitive nature of industry does not allow a high degree of on the job training once provided by industry and the better prepared students can enhance industry's productivity by minimization secondary training needed to bring up newly graduated employees to a useful form. In this regard, the courses offered graduate students with skill sets needed to identify corrosion problems in oil and gas industry and have needed training in analytical characterization tools and procedures such that once they join task force they will require minimal extra training and are enabled to perform failure analysis and data interpretation skills to minimize material loss hence enhancing productivity and in preventing failures that could potentially cost human lives. Through this course members of student groups in each project worked together to follow 14 steps in failure analysis. Some steps are more trivial but critical such as sample collection, information gathering, etc. But other steps requires utilization of advanced instrumentation and in depth data analysis that requires highly skilled personnel. Each student took responsibility of certain aspects of the failure analysis steps and each week students performed materials characterization techniques such as metallography, mechanical testing, optical microscopy, scanning electron microscopy, energy dispersive spectroscopy, and Fourier transform infrared spectrophotometry. Finally toward the end of the semester, each team met in groups and brainstormed on their findings and contributed to the analysis of data and conclusions made for each case studies.

At the end of the semester each group provided the whole class with a comprehensive technical presentation showing all their results and analysis and students learned quite a bit from each other's experiences.

In such a course a challenge has been and will always be to find an industry and their personnel who are dedicated to education and willing to go out of their way to help educational program by providing actual field samples and corresponding historical background so that students get the benefit of having as much historical background information. It is conceivable that most industries have personnel changes during the years due to retirements and other reasons and no perfect historical data is available in many cases. However, good bookkeeping and willingness of the industries in this regard will be an asset in success of such teaching experiences.

Overall, the course was a success and students who signed up for the course were very satisfied as indicated by Student Perception of Teaching (SPOT) survey delivered by university administration at the end of the semester. Results of SPOT survey is provided in the Appendix.

## 6. Summary

Several benefits of having practical course content in a graduate level Engineering Technology course includes:

1. A stronger collaboration between university and industry is facilitated in such interactions that benefits both industry and the educational institution.
2. Graduate students gain a highly valuable experience is analyzing failed components from actual field that enhances their learning experience and keep them motivated in technical fields.
3. Graduate students in such a course are exposed to advanced analytical techniques such as electron microscopy, energy dispersive spectroscopy, x-ray diffraction, Fourier transform infrared spectrophotometry to name a few. Skills in running such instruments and being able to interpret data generated in such techniques can prove valuable in landing gainful jobs.
4. Through authorship sharing of such articles students gain experience and confidence that motivates them to apply what they have learned in the failure analysis course to problems they are tackling in their own thesis/dissertation.

## Acknowledgement

All samples used in this study were provided by EOG Resources. All authors are highly appreciative of Mr. Chris Ezell who gathered all field samples and providing historical background for each sample.

## References

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Fig. 2 Failed oil separator weir plate.

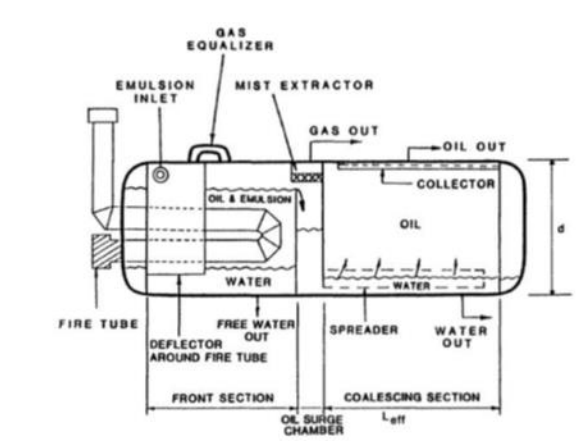


Fig. 1 Schematic of a separator system used in oil and gas

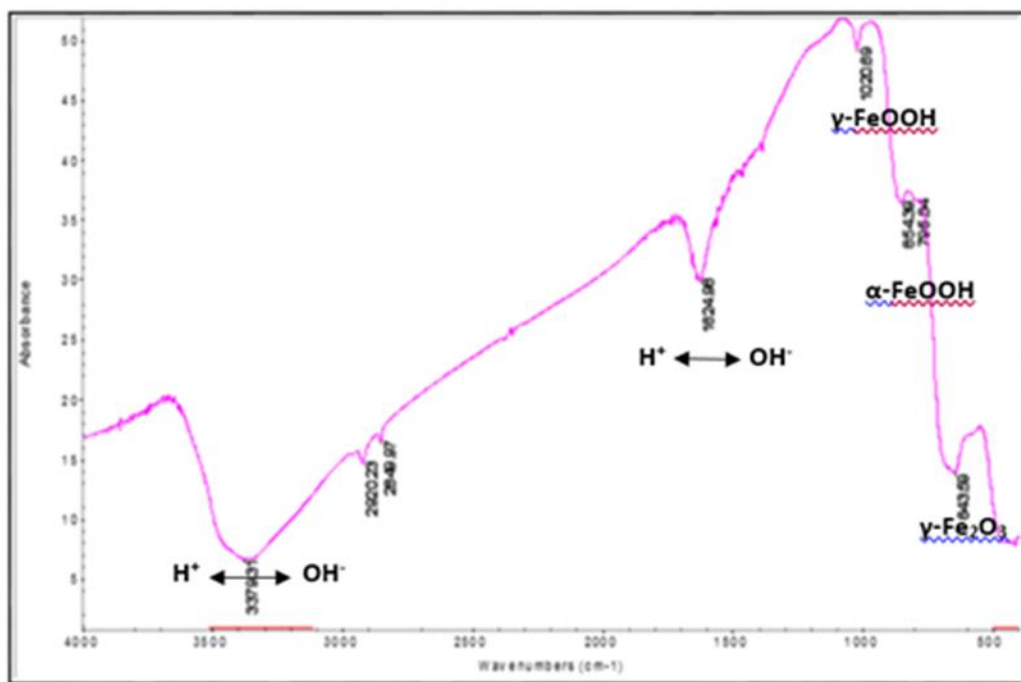


Fig. 3: FTIR spectrum of corrosion product form on the weir plate showing presence of  $\gamma\text{-Fe}_2\text{O}_3$ ,  $\alpha\text{-FeOOH}$ , and  $\gamma\text{-FeOOH}$  as the main rust constituents.



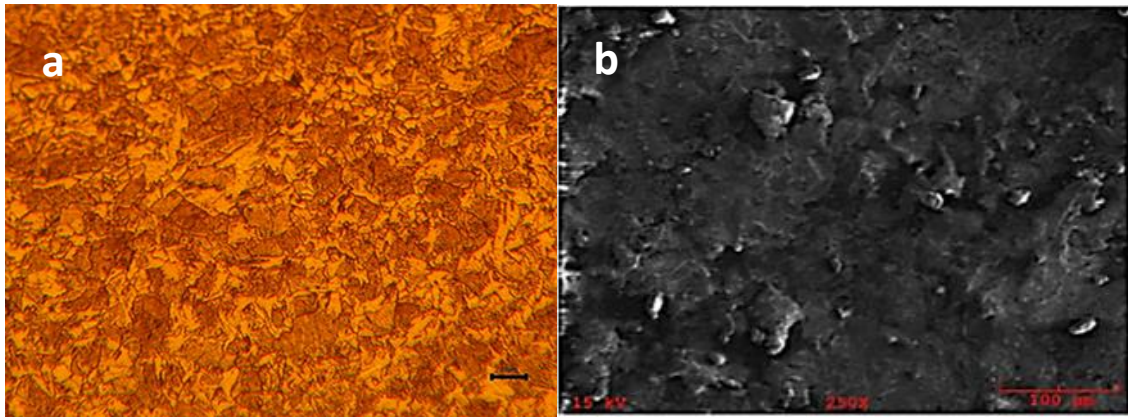


Fig. 4 Optical micrograph (a) and SEM micrograph (b) of a severely corroded weir plate from an operational oil exploration

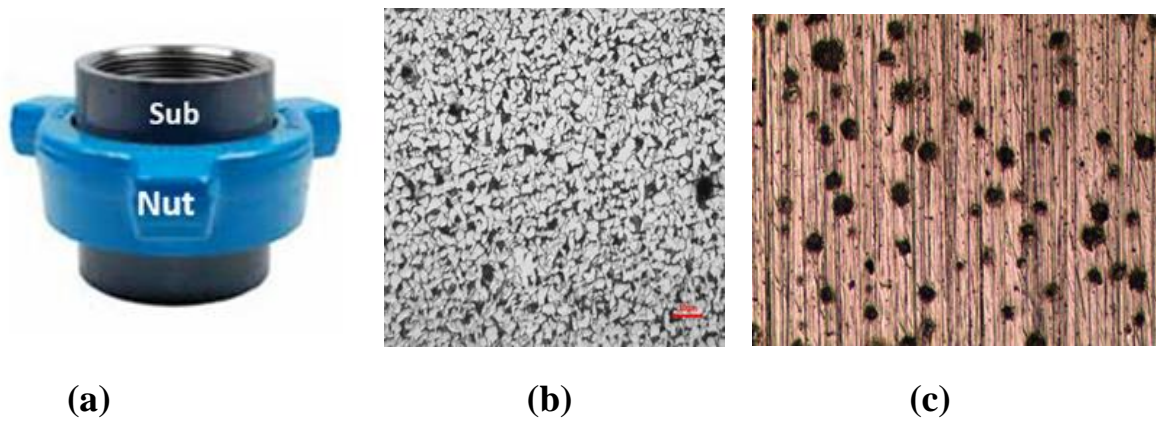


Fig. 5: Hammer union system (a), microstructure of the sub showing pearlite phase (b), and microstructure of the nut showing nodules in nodular cast iron (c).



Fig. 6: External part of a API-5L Grade B low carbon steel pipe (a) and corresponding internal image (b).

## Appendix

MSET 5150 001	Evaluation Delivery: Online
Applications of Electron Microscopy and Failure Analysis	Evaluation Form: B
Course type: Face-to-Face	Responses: 10/11 (91% very high)
Taught by: Seifollah Nasrazadani	
Instructor Evaluated: Seifollah Nasrazadani-Professor	

**Overall Summative Rating** represents the combined responses of students to the four global summative items and is presented to provide an overall index of the class's quality:

Median  
4.7  
(0=lowest; 5=highest)

**Challenge and Engagement Index (CEI)** combines student responses to several *IASystem* items relating to how academically challenging students found the course to be and how engaged they were:

CEI: 6.1  
(1=lowest; 7=highest)

### SUMMATIVE ITEMS

	N	Excellent (5)	Very Good (4)	Good (3)	Fair (2)	Poor (1)	Very Poor (0)	Median
The course as a whole was:	9	44%	33%	11%		11%		4.3
The course content was:	9	56%	22%	11%		11%		4.6
The instructor's contribution to the course was:	9	78%		11%		11%		4.9
The instructor's effectiveness in teaching the subject matter was:	9	78%		11%		11%		4.9

### STUDENT ENGAGEMENT

	N	Much Higher (7)	(6)	(5)	Average (4)	(3)	(2)	Much Lower (1)	Median
Relative to other college courses you have taken:									
Do you expect your grade in this course to be:	10	40%	40%	20%					6.2
The intellectual challenge presented was:	10	50%	30%	20%					6.5
The amount of effort you put into this course was:	10	50%	30%	20%					6.5
The amount of effort to succeed in this course was:	10	60%	30%	10%					6.7
Your involvement in course (doing assignments, attending classes, etc.) was:	10	50%	40%	10%					6.5

### STANDARD OPEN-ENDED QUESTIONS

**Was this class intellectually stimulating? Did it stretch your thinking? Why or why not?**

1. yes it did
2. yes. got to know many new things
3. This provided a fantastic explanation of fatigue/creep/corrosion failure and how to find it and prevent it. I know how some advanced lab equipment functions now, and had an opportunity to advance my academic status through possible publication.
4. Yes it was stimulating. It did stretch my thinking. We had a big project. You need critical thinking to finish your project.
5. Yes, the material thought was very interesting
6. Nothing

**What aspects of this class contributed most to your learning?**

1. the slides and insights given by the professor
2. project
3. It went perfectly with the Fatigue and Creep course. From the other course I learned more about how it works. From this course I learned about how to recognize it using lab equipment and what to do about it.
4. The project and Course content.
5. The discussion style of the class was a good way of learning
6. Project

**What aspects of this class detracted from your learning?**

1. none
2. nothing
3. scheduling of assignments left some pretty severe crunch-time near the end...
4. Nothing
5. I feel the dead lines were a bit tough. Some projects were given too late in the semester, and could've been given earlier and more time to prepare for it would've been awesome
6. Research

**What suggestions do you have for improving the class?**

1. more information
2. nothing
3. This is one of the best classes I've ever taken, and I want to learn more about this subject.
4. Nothing
5. Just giving projects with more time in advance
6. more homework and more project
7. Start the failure analysis project earlier in the semester.